# PATENT ABSTRACTS OF JAPAN

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## 54) METHOD FOR MANUFACTURING SUBSTRATE FOR MAGNETIC RECORDING MEDIUM

57)Abstract:
'ROBLEM TO BE SOLVED: To provide a method of manufacturing a substrate for a magnetic recording medium aving a NiP electroless plating layer with high adhesion to a glass substrate.
OLUTION: After a glass substrate surface is lapped by using a rubbing liquid composition containing silicon arbide particles or alumina particles a maximum particle size of which is 130 μm or smaller, a particle size is 110 m or smaller at a 3% cummulative height, and a 0.5 μm or larger at a 94% commulative height, it is subjected to hemical etching. And then, it is subjected to NiP electroless plating.

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LAIMS

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:laim(s)]
:laim 1] The manufacture method of the substrate for magnetic-recording media which a maximum droplet size is 30 micrometers or less, and is characterized by performing chemical etching processing and subsequently performing iP electroless deposition processing on it after the particle size in 3% of accumulation height performs lap processing ı a glass-substrate front face using the polish liquid constituent with which the particle size in 110 micrometers or less ıd 94% of accumulation height contains a silicon-carbide particle or an alumina particle 0.5 micrometers or more.
:laim 2] The manufacture method of the substrate for magnetic-recording media according to claim 1 that a glass ıbstrate is characterized by the bird clapper from SiO2-Li2 O system glass ceramics.

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NOTICES *



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**** shows the word which can not be translated.
In the drawings, any words are not translated.

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ETAILED DESCRIPTION

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)etailed Description of the Invention]
'001]
'he technical field to which invention belongs] this invention relates to the manufacture method of the glass substrate ·r magnetic-recording media. Furthermore, it is involved in the manufacture method of a glass substrate and the ıbstrate for magnetic-recording media which has a NiP electroless deposition layer with high adhesion. Specifically, it related with the manufacture method of the substrate in high recording density magnetic-recording media, such as a in film magnetic-recording disk of the cover half used by the information industry etc.
'002]
)escription of the Prior Art] In recent years, many hard disk equipments are used as external storage of information ·ocessors, such as a computer. After forming a NiP electroless deposition layer in the front face of the nonmagnetic ıbstrate which consists of an aluminium alloy generally and performing necessary data smoothing, texture ring ·ocessing, etc., the magnetic disk carried in this hard disk equipment forms a non-magnetic metal ground layer, a agnetic layer, a protective layer, a lubricating layer, etc. one by one on it, and is produced.
'003] In the magnetic disk unit, although the head for record reproduction is moving by the fixed flying height in the agnetic-recording medium top, this flying height is very small with the rapid increase in the field recording density of magnetic-recording medium in recent years. Moreover, in order for the miniaturization of a magnetic disk unit and ghtweight-ization to also progress quickly and to correspond to these, it is required to make still smaller granularity of e front face of a magnetic-recording medium, and medium surface roughness is already small to about :veralangstroms by Ra. Furthermore, since the shock resistance required of a magnetic disk since it corresponds to )rtability type hard disk equipment is also becoming 400G-800G, and a high value, in the substrate which consists of e conventional aluminium alloy to shock resistance, correspondence is difficult. Then, the glass substrate which can tain very small surface roughness and is excellent also in the mechanical strength instead of the aluminium alloy ıbstrate from standpoints, such as shock resistance and surface smooth nature, is beginning to be used.
'004] In many cases, in the aluminium alloy substrate which gave NiP electroless deposition, the concentric circle-ce texture ring is given to the substrate circumferencial direction by polish on the front face. If good, this mainly oses the friction property between the head for record reproduction, and a magnetic-recording medium, and aims at :curing endurance. Moreover, in recent years, to replace with the texture ring by polish and to form a salient only in a SS zone with the texture ring by the laser beam, i.e., a laser beam, in connection with the flying height of the head at e time of a magnetic-disk-unit operation being remarkably small, is tried. (JP,8-129749,A etc.)
'005] However, since the salient configuration controllability is bad, it is very difficult unlike the aluminium alloy ıbstrate which gave NiP electroless deposition, to irradiate a direct laser beam and to form a salient in a glass plate. hen, in order to apply laser texture technology to a glass substrate, it is necessary to form a NiP electroless deposition yer on a substrate beforehand.
'006] Forming a NiP electroless deposition film on a glass substrate is proposed by JP,61-54018,A. However, it is chnically difficult to form a NiP layer in a glass substrate with sufficient adhesion by the electroless deposition ethod. Then, in order to improve the adhesion of a glass substrate and a NiP electroless deposition film, the method split-face-izing mechanically or chemically the glass-substrate front face used for plating and the method of erforming pretreatment of electroless deposition are proposed. for example, -- as the mechanical split-face-ized ethod -- aluminum 2O3 etc. -- the method which 100A or more of surface roughness grinds by Ra in center line verage coarseness by the grinding stone using the abrasive material is learned, and as the chemical ****-ized method, ter carrying out alkaline degreasing, the method of **********ing by the hydrofluoric acid etc. is learned Moreover, e method continuously carried out sensitization and activated with the solution of a palladium chloride with the )lution of a stannous chloride as pretreatment of electroless deposition is proposed. (JP,7-272263,A etc.)

)07]
roblem(s) to be Solved by the Invention] However, by these methods, the NiP layer which has sufficient adhesion d sufficient smooth nature to obtain a good magnetic disk was not able to be formed by the electroless deposition ethod on the glass substrate. this invention is made in view of an above-mentioned point, and the purpose is excellent the adhesion of a glass substrate and a NiP electroless deposition layer, and it has high shock resistance and surface ıooth nature, and is in moreover offering the manufacture method of the substrate for magnetic-recording media that ɜ low surfacing height of a head is obtained by being stabilized.
)08]
leans for Solving the Problem] As a result of inquiring wholeheartedly in view of the above-mentioned actual ndition, by performing wrapping processing which used the specific polish abrasive grain for the glass substrate, this vention persons find out that the outstanding NiP layer which satisfies many above-mentioned requirements is rmed on a substrate, and reach this invention. That is, after the summary of this invention performs lap processing on glass-substrate front face using the polish liquid constituent containing a silicon-carbide particle or an alumina rticle with the maximum droplet size of 130 micrometers or less, a particle size [ in 3% of accumulation height / of 0 micrometers or less ], and a particle size [ in 94% of accumulation height ] of 0.5 micrometers or more, it performs emical etching processing and consists in the manufacture method of the substrate for magnetic-recording media aracterized by subsequently performing NiP electroless deposition processing.
009] Hereafter, this invention is explained in detail. After carrying out lap processing of the front face of a glass bstrate, a detailed crevice is made to form in a substrate front face by carrying out chemical etching, in order that the bstrate for magnetic-recording media of this invention may raise the adhesion of a glass substrate and a NiP ectroless deposition layer. As a glass substrate, although especially the quality of the material is not limited, glass ramics are desirable and the glass ceramics of a SiO2-Li2 O system are used further suitably. Without spoiling rface smooth nature to some extent, since the amorphous field on the front face of a substrate can be alternatively ched if glass ceramics are used, since this can form a detailed crevice appropriately, it is suitable. When the degree of ystallization is small, a detailed depression is not formed of uniform etching, but adhesion tends to get worse by it.
010] The polish liquid constituent used for lap processing of this invention contains a silicon-carbide particle or an umina particle as an abrasive grain, and particle size [ in / 110 micrometers or less and 94% of accumulation height / the particle size in 130 micrometers or less and 3% of accumulation height ] is required for the maximum droplet ze of this abrasive grain, when that it is 0.5 micrometers or more raises the adhesion of a glass substrate and a NiP posit. Each of maximum droplet sizes of these abrasive grains, 3% of accumulation height, and particle size in 94% JIS by the electric resistance examining method. R It asks by measuring by 6111. Moreover, it is desirable to use the ing containing a surfactant as a polish liquid constituent of this invention.
)011] Lap processing performed using the polish liquid constituent of this invention can be performed by the nventional method. As a typical method, the method of grinding a glass-substrate front face etc. is mentioned, for ample, supplying the polish liquid of this invention to an abrasive cloth or polish putt, after performing thickness ljustment of glass by the grinding processing by the bonded abrasive. Although the glass substrate used does not need be carried out even if the mirror finish of the front face is beforehand carried out by polish processing, in industrial oduction, the method of not performing polish processing in the meaning which reduces a process is suitable for it.
)012] As for the glass substrate obtained by lap processing by this invention, it is desirable that center line surface ughness is 0.01-1.0 micrometers, and further 0.01-0.5 micrometers is suitable for it. This configuration contributes to rmation of the detailed crevice formed by the chemical etching processing performed at a next process greatly. The lass substrate used for this invention has desirable glass ceramics, and it is suitable to use SiO2-Li2 O system glass eramics especially.
)013] Subsequently chemical etching processing is performed to the glass substrate which performed lap processing. s an etching reagent used for chemical etching, fluoric acid system etching reagents, such as fluoric acid, a potassium uoride, an ammonium fluoride, and an acid ammonium fluoride (NH4 F-HF), are desirable. This will depend glass eramics on the portion which **********s comparatively alternatively and the amorphous (amorphous-izing) portion glass ceramics is crystallizing tending [ comparatively ] to remain, if etching processing is performed by the fluoric cid system etching reagent. It is thought that this portion that remained becomes the detailed crevice which brings bout the anchor effect which was excellent when NiP electroless deposition was performed by this.
)014] The size of this detailed crevice can be controlled by choosing suitably the concentration of an etching reagent, rocessing temperature, the processing time, etc. According to this invention, the glass substrate which has the detailed revice whose width of face the length of a crevice is 4-20 micrometers, and is 1-5 micrometers on a front face can be btained by lap processing and chemical etching processing. When the length and width of face of a crevice exceed the ase of under the above-mentioned range, or this range, adhesion with a NiP electroless deposition layer is not obtained

ɔugh. Here, the length of a crevice indicates a part for the longest bay in a direction perpendicular to the length to be dth of face for a part for a longest crevice bay. Moreover, when the length of each crevice differs from width of face, the 20 or more averages be the length of a crevice, and width of face.

)15] Moreover, it is suitable for the aspect ratio of a crevice that it is 0.3-0.7. Here, an aspect ratio shows the ıximum width of a hole, and the ratio (maximum width / the maximum length) of the maximum length. NiP seldom mes to enter at the time of plating as an aspect ratio is less than 0.3. Moreover, an anchor effect becomes weak and is t desirable if 0.7 is exceeded. Furthermore, when it observes by 600 times of SEM, as for the rate of area of a ɔvice, it is desirable that it is 0.5 - 50% to a substrate front face. The adhesion of plating will tend to become weak if ɔ rate of area exceeds less than 0.5% and 50%.

)16] The crevice on these front faces of a substrate is observed and measured as a black portion with the secondary ɔctron image of a scanning electron microscope (SEM). That is, since a shadow is hit when it observes by the ɔondary electron image of SEM, a crevice is observed black. Specifically, using SEM 600 times the scale factor of .s, the length and width of face of a crevice lean a substrate front face 40 degrees to the detector of a secondary ɔctron line, observe it, and are measured.

017] The substrate for magnetic-recording media obtained by this invention is manufactured through a susceptibility-ɔd process, an activation process, and a NiP electroless deposition process one by one by the well-known method. ıd before susceptibility processing, degreasing processing is usually prepared. Moreover, it is good to establish a ısing process between each process, to use ion exchange water or ultrapure water suitably as a wash water, and to ck in a wash bath suitably.

018] A degreasing process is a process which washes the front face of a glass substrate, for example, the method of ing ultrapure water, an alkali cleaner, an acid cleaning agent, a surfactant, etc. is mentioned. A susceptibility-ized ocess and an activation process are processes which give catalytic activity required in order to make a glass substrate ırt NiP electroless deposition. That is, a glass front face needs to form the catalyst nucleus of noble metals, such as u, Pt, Pd, and Ag, on the surface of glass, in order to start electroless deposition, since there is no catalytic activity.

019] Each above-mentioned process is carried out as follows by the well-known method. The divalent metal ion hich consists of Sn, Ti, Pd, Hg, etc. is made to adsorb first in a susceptibility-ized process. Usually, it is used suitably, ıd in ordinary temperature, the tin chloride solution of about 0.05 g/l is immersed about 2 minutes into tin chloride ılution, and rinses. Next, a catalyst nucleus is made to form in the included activation solution containing the noble etals which serve as the aforementioned catalyst nucleus as an activation process on the surface of a glass substrate / reduction operation of the divalent metal ion which was immersed and adsorbed the above-mentioned glass ıbstrate. Usually, it is used suitably, and in ordinary temperature, the palladium-chloride solution of about 0.05 g/l is ımersed about 2 minutes into palladium-chloride solution, and rinses. A susceptibility-ized process and an activation ·ocess are good also as the same process by using the mixed-water solution of tin chloride and a palladium chloride.

)020] NiP electroless deposition of the glass substrate processed at the activation process is carried out by the well-ıown method. Usually, a commercial NiP electroless deposition bath is used and predetermined-time processing of .e glass substrate is carried out in a plating bath. Although NiP electroless deposition layer thickness is chosen ·bitrarily, for a good magnetic-recording medium, the range of 1-10 micrometers is good.

)021] According to this invention persons' knowledge, in order to raise the adhesion of a glass substrate and a NiP .yer, it is required to heighten a physical anchor effect and it is effective. That is, the length which was formed in the ıbstrate front face by chemical etching processing according to this invention is considered that 1-5 micrometers ıters [ 3-20 micrometers and width of face ], Sn enters [ an aspect ratio ] by reception-ized processing, and Pd enters ı a reduction operation by activation further in the detailed crevice of 0.3-0.7. Therefore, since a NiP film is formed ıto this detailed hole in case a NiP film is formed by NiP plating processing, a physical anchor effect is heightened ıd it is thought that adhesion with a glass substrate and a NiP deposit is strengthened by this. If needed, polish rocessing can be performed or the glass substrate which gave NiP electroless deposition can perform suitably texture rocessing of the texture ring by the laser beam, a machine texture ring, etc.

)022] Subsequently, a magnetic-recording layer is formed according to a conventional method. Usually, it forms so ıat the laminating of each class may be carried out to the order of Cr ground layer, a magnetic layer, a protective ıyer, and a lubricating layer. Although the thickness of Cr ground layer is set as a magnetic-recording medium ccording to desired magnetic properties, it is usually 100-1000A. Cr ground layer is usually pure -- although formed y Cr, you may make other elements to a 20 atom % grade contain in total Although there is usually one Cr ground ıyer, if it is a request, also let it be the multilayer which consists of two or more layers.

)023] A magnetic layer is usually formed by Co system alloy, for example, CoNiCr, CoCr, CoCrTa, CoCrPt, :oCrPtTa, CoCrPtB, CoNiPt, CoNiCrBTa, CoSm, etc. The thickness of a magnetic layer is usually 100-500A. You ıay be a multilayer even if the number of magnetic layers is also one. A protective layer is usually formed by metallic

des, such as carbon materials, such as amorphous carbon and hydrogenation carbon, and a silica, a zirconia, and 30-
)A of the thickness is usually 30-200A preferably. You may be a multilayer even if the number of protective layers
ılso one.
)24] A lubricating layer is formed by applying a fluorine system fluid lubrication agent etc. to a protective layer. In
ılition, although a protective layer and a lubricating layer are not indispensable as a magnetic-recording medium, it is
:y desirable [ a lubricating layer ] to prepare both this layer, considering the endurance of a magnetic-recording
:dium, a friction property with the head for record reproduction, etc. Formation of a ground layer, a magnetic layer,
ł a protective layer can be performed by conventional methods, such as a DC-sputtering method, a RF-sputtering
:thod, and a vacuum deposition method. According to this invention, by performing NiP electroless deposition to the
ɔve glass substrates, it has the adhesion of sufficient strength which does not cause exfoliation with a glass substrate
ł a NiP electroless deposition film etc., and it becomes possible to obtain the magnetic-recording medium excellent
shock resistance.
)25]
xample] Hereafter, although an example explains this invention still in detail, this invention is not limited to the
.lowing examples, unless the summary is exceeded. In addition, in each example, measurement and evaluation were
rformed on condition that the following.
)26] (1) Using the scanning electron microscope (SEM) 600 times the surface type-like scale factor of a glass
bstrate of this, it leaned 40 degrees, observed to the detector of a secondary electron line, and asked for the rate of
:a of the crevice to the length and the width of face, aspect ratio, and substrate front face of a crevice on the front
:e of a substrate.
) Adhesion JISK5400 of a glass substrate and a NiP electroless deposition layer The cross cut adhesion test of 8.15
timated adhesion. It is shown that the evaluation mark 10 have good adhesion.
) With the surface roughness meter (KEERUE ten call company make P-12) which has the sensing pin whose nose of
m is phi0.2micrometer, surface roughness center line surface roughness (Ra) was performed by 240 micrometers of
easurement length, and calculated and evaluated the average.
027] Glass ceramics of the SiO2-Li2 O system of example 1 marketing. After using it and performing grinding
liding) processing by the bonded abrasive, it wrapped by grain-size partition #1000 (less than [ maximum-droplet-
:e 27micrometer ], particle size of 23 micrometers or less of 3% of accumulation height, particle size of 5.0
icrometers or more of 94% of accumulation height) of the artificial abrasives F0 (a compound artificial emery, 3.90
more specific gravity, more than aluminum2 03:45 % of the weight, less than [ TiO:2.0 % of the weight ], less than
ZrSiO:49 % of the weight ]) made from FUJIMIINKOPORE. The center line average surface roughness (Ra) of the
)tained substrate was 0.3 micrometers.
028] Next, it rinsed after the washing processing for 10 minutes at 50 degrees C of bath temperature by the alkaline
eaning agent for glass ( PK-LCG22 by , Inc. Parker), and rinsed by immersing for 2 minutes and the above-
entioned glass ceramics at a room temperature, and subsequently to the inside of acid ammonium-fluoride ( NHby
anto chemistry incorporated company4 F-HF JIS number K8817) 50 g/l, performing etching processing.
029] The length of the detailed crevice on the obtained front face of a glass substrate was 4.9 micrometers, width of
ce was 2.9 micrometers, and 0.59 and the rate of area of the aspect ratio of a crevice were 13.4%. Next, it is the glass
ıbstrate which has a detailed crevice SnCl2 of commercial 0.05 g/l It rinsed by having been immersed in solution for
minutes at the room temperature, and susceptibility-ized processing was performed. Then, PdCl2 of commercial 0.05
l It rinsed by having been immersed in solution for 2 minutes at the room temperature, and activation was performed.
ıbsequently, the NiP layer of 15 micrometers of thickness was formed by NiP electroless deposition. Furthermore,
ıking processing of 1 hour was performed at 150 degrees C. Thus, as a result of evaluating the adhesion of the
)tained NiP layer and a glass substrate, evaluation mark are 10 and having good adhesion was checked.
)030] Abrasive material FO#1000 of example 2 example 1. instead of using it -- FO (the compound artificial emery
ıade from FUJIMIINKOPORE --) 3.90 or more specific gravity, aluminum 2O3 : 45 % of the weight or more, less
ıan [ TiO:2.0 % of the weight ], ZrSiO: Except having wrapped by grain-size partition #2000 not more than 49 % of
ıe weight (15 micrometers or less of maximum droplet sizes, particle size of 14 micrometers or less of 3% of
:cumulation height, particle size of 2.0 micrometers or more of 94% of accumulation height), it is the same method as
ı example 1, and the NiP electroless deposition layer was formed in the glass substrate. The centerline surface
)ughness after lap processing, the configuration of the detailed crevice after etching processing, and the adhesion of a
lass substrate and a NiP electroless deposition layer are shown in Table 1. All are 10 and the evaluation mark of
ılhesion have good adhesion for them.
)031] Instead of using abrasive material FO#1000 of example 3 example 1, except having wrapped by GC#3000 (the
roduct made from FUJIMIINKOPORE, 96 % of the weight or more of SiC(s), 13 micrometers or less of maximum

oplet sizes, particle size of 11 micrometers or less of 3% of accumulation height, particle size of 2.0 micrometers or ore of 94% of accumulation height), it is the same method as an example 1, and the NiP electroless deposition layer as formed in the glass substrate. A result is shown in Table 1.

032] Instead of using abrasive material FO#1000 of example of comparison 1 example 1, except having wrapped ith cerium-oxide polish liquid (speed FARM company make, 10 micrometers or less of maximum droplet sizes, rticle size of 0.24 micrometers or less of 3% of accumulation height, particle size of 3.0 micrometers or more of 94% accumulation height), it is the same method as an example 1, and the NiP electroless deposition layer was formed in e glass substrate. A result is shown in Table 1. The evaluation mark of adhesion are 5 and were not able to obtain fficient adhesion.

033]

able 1]

---------- Center line Concave Section Adhesion Surface roughness Length Width of face Aspect tio Rate of area Evaluation mark (micrometer) (micrometer) (micrometer) (%)

---------- ---------- example 1. 0.3 4.9 2.9 0.59 13.4 10 Example 2 0.05 3.4 2.1 0.62 2.7 10 Example 3 0.09 1 1.5 0.48 1.3 10 Example 1 of comparison 0.0005 3.4 3.3 0.97 0.3 5 ----------.

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